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Studies on the Effect of Mono-Alkoxy Pyrophophato Coupling Agent on the Mechanical Properties of Magnesium Hydroxide Filled HDPE/EVA/$Mg(OH)_2$/Composites

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ABSTRACT

Effect of treatment of coupling agent (mono-alkoxy pyrophophato coupling agent) on mechanical properties of composites made from HDPE/EVA/$Mg(OH)_2$/ Composites is reported here. The coupling agent in the form of solution (1.5%) was used for treatment of the filler .The treatment resulted in enhancement of mechanical properties of composites when compared with composites containing untreated Magnesium hydroxide. The properties under consideration were tensile strength, modulus at (%) elongation at break, elastic modulus, hardness, etc. Although good reinforcement was observed due to treatment of 1.5% coupling agent, observed was very much remarkable by its comparing to untreated once. Comparison of properties of composites filled with treated and untreated Magnesium hydroxide established that the treatment of Magnesium hydroxide imparts better reinforcing properties. The properties under consideration were tensile strength, (%) elongation at break, elastic modulus, hardness, etc. Tensile strength was improved by 19.10%, (%) elongation at break was improved by 9.01%, elastic modulus was improved by 100%, while hardness was improved by 0.5%, at (0.14) volume fraction.

Keywords: Titanate Coupling Agent, Mechanical Properties of Magnesium hydroxide, HDPE/EVA/$Mg(OH)_2$/TCA-114.

INTRODUCTION

Many engineering polymer materials are multiphase systems. Polymer blends are physical mixtures of structurally different homo- or copolymers. Polymer alloys may be considered as a subclass of polymer blends and is a term that describes multiphase polymer systems with a modified interface [1]. The addition of fillers can significantly change the mechanical characteristics of a material. However the existence of fillers is sometimes essential for maintaining the mechanical integrity of a material. Some of these additives, such as the coupler/coupler solvent dispersions or the latex, were prepared in solutions and could not be easily extracted to form solid films for evaluation [2]. Coupling agents are additives used in reinforced and filled plastic composites to enhance the plastic–filler-reinforcement interface to meet increasingly demanding performance requirements. In general, there is little affinity between inorganic materials used as reinforcements and fillers and the organic matrices in which they are blended. With silicate reinforcements, silane coupling agents act by changing the interface between the dissimilar phases. This results in improved bonding and upgraded mechanical properties [3].

Magnesium hydroxide as a filler play a key role in modifying and enhancing the mechanical properties of rubber, ceramic, paint, plastic and other industries [4-5].

It is reported [6-8] that titanate coupling agents are considered useful as promoters of adhesion between mineral fillers and organic matrix. These additives provide improved mechanical strength as well as chemical resistance to composites. the Titanate coupling agent with the appropriate functionality provide chemically bonded coupling agent between this mineral filler particles and the plastic network and is responsible for the improved reinforcing action of mineral fillers.

In this work the effect of titanate coupling agent on mechanical properties of composites made from and Magnesium hydroxide (treated and untreated) has been studied. Treated $Mg(OH)_2$ is prepared by mixing 1.5 gram of titanate with 100 g of $Mg(OH)_2$, while untreated does not content coupling agent. Composites of HDPE/EVA with treated and untreated $Mg(OH)_2$ were prepared in various loadings . The magnitudes of properties of composites containing treated and untreated filler are compared.

Tensile strength, (%) elongation at break, elastic modulus, hardness, etc. are used to study the mechanical properties of prepared polymer composites.

EXPERIMENTAL

Materials

High-density polyethylene (HDPE, 5502#, MFR=0.35g/10min) was from Daelim, Korea, Ethylene vinyl-acetate copolymer (EVA, 8450#, VA=15%wt, MFR=1.5g/10min) was from Nippon, Japan. The filler magnesium hydroxide ($Mg(OH)_2$, average particle size=2μm) was obtained from Dalian Yatai Science and Technology New Material Co.Ltd. , China, Titanate coupling agent [(TCA-114)] was obtained from Anhui Tianchang Organic Chemical Plant is the organotitanium pilot base of Shanghai Institute of Organic Chemistry.

General characteristics Ethylene vinyl-acetate copolymer, general Characteristics of High Density Polyethylene, Physical characterization of Titanate coupling agents (TCA-114), General Properties of Magnesium hydroxide, are reported in table 1, 2 and 3 respectively.

Table (1): General Characteristics of Ethylene vinyl-acetate copolymer.

Trade Name	NUC 8450 , Nippon Unicar Co ,Ltd
Appearance	White
Mooney Viscosity (100^0C)	40
Specific Gravity (g/cm^3)	0.94
Melt index (g/10 min)	0.1 -10
Ash Content (%)	0.3

Table (2): General Characteristics of High Density Polyethylene.

Trade Name	HDPE, 5502 **,** Daelim, Korea
Appearance	White
Specific Gravity (g/cm^3)	0.96
Melt index (g/10 min)	0.05-0.8

Table (3): Physical characterization of Titanate coupling agents (TCA - 114).

Chemical Name	mono-alkoxy pyrophophato coupling agent
Typical purity (%) Physical form Color	99 Liquid light yellow viscous liquid
Density	(GB4472-84) D30 About 1.03g/cm3
Flash point (^{0}c)	(GB37-77)open 50
Refractive index	(GB6428-86)ND30 About 1.45
Viscosity (cp)	(GB265-70) 30 About 300mm/s
pH	3
Solubility	Isopropyl alcohol, xylene, Toluene, DOP, Mineral oil, MEK .

Table (4): General Properties of Magnesium hydroxide .

Name	Magnesium hydroxide
Molecular formula	Mg(OH)
Molar mass (g/mol)	58.33
Appearance	White solid
Melting point (°C)	Decomposes at 623 K (350 °C)
Density (g/cm³)	2.4
Solubility	0.0012 g in 100 g water
pH	6.5-7

Treatment on Magnesium hydroxide by Titanate Coupling Agent

The coupling agent (1.5g) was mixed [9-13] with isopropyl alcohol (100 ml) to make a solution for applying to filler (100 g). 1.5 grams of coupling agent was used per 100g of Magnesium hydroxide. The filler (Magnesium hydroxide) was mixed with the solution of coupling agent in isopropyl alcohol with stirring to ensure uniform distribution of the coupling agent, mixing continued for 30 minutes. The treated filler (Magnesium hydroxide) was then dried at 100 ^{0}C in an oven for about 6 hours to allow complete evaporation of the alcohol.

Preparation of composites

EVA/HDPE/$Mg(OH)_2$/TCA-114 composites were prepared via melt compounding at 160 ^{o}C in ThermoHaake rheomixer with a rotation speed of 60 rpm, and the mixing time is 6 min for each sample. The mixed samples were transferred to a mold and preheated at 180 ^{o}C for 15 min, then pressed at 20 MPa and then successively cooled to room temperature while maintaining the pressure to obtain the composites sheets for further measurements. Before mixing, all the components were dried in vacuum oven at 80 ^{o}C for at least 12h.

Table (5): Compounding Recipe For HDPE/EVA/$Mg(OH)_2$ / TCA-114 .

Volume Fraction of Composites	EVA /HDPE (Wt – g)	Mg(OH)2 (Wt – g)
0.00	25/25 =50	0.0
0.04	22.5/22.5 =45	5
0.09	20/20 =40	10
0.14	17.5/17.5 =35	15
0.21	15/15 =30	20
0.28	12.5/12.5 =25	25
0.37	10/10 =20	30
0.48	7.5/7.5 =15	35
Filler (Treated &Untreated)	Variable (0.0 – 0.48 Volume fraction)	
Curing Time (min)	15	
Curing Temp (^oC)	180	

Scanning electron microscopy (SEM)

The SEM micrographs of samples were observed by JEOL JSM-5510 scanning electron microscope. The samples are chosen after the tensile test. The content of HDPE/EVA/$Mg(OH)_2$ at 0.48 Volume fraction . The surface of the treated and untreated samples was coated with a thin layer of gold to avoid electrostatic charging during examination. Photographs of representative areas of the sample were taken at 5000X magnifications**.**

Measurement of Mechanical Properties

Mechanical properties such as tensile strength, elongation at break, elastic modulus were determined by subjecting dumbbell shaped specimens (in confirmation with ASTM D – 638) to a universal testing machine (Shenzhen Reger Instrument Co. Ltd, China). The sheets from which specimens were cut had been conditioned for 24 hours prior to subjecting to universal testing machine (100 kg load cell), at a crosshead speed of 50 mm / min. Hardness was measured on Machine –LX–A ,produced by Shanghai , Liuzhong meterage , factory .

RESULTS AND DISCUSSION

Tensile strength

The dependence of the tensile strength on volume fraction of magnesium hydroxide is represented in figure 1. It is seen that on increasing the volume fraction of (both treated and untreated) magnesium hydroxide , the tensile strength increases up to a certain value and it declines .The peak values of tensile strength of the composites correspond to 12.5 MPa and 10.5 MPa for treated and untreated Magnesium hydroxide composites respectively. It is noteworthy that the tensile strength of composites filled with treated Magnesium hydroxide at 0.14 volume fraction is 1.19 higher than that of untreated Magnesium hydroxide composites.

Modulus at (%) elongations at break

The dependence of modulus at (%) elongation at break with volume fraction of treated and untreated HDPE/EVA/$Mg(OH)_2$ Composites is depicted in Fig. 2. In the cases modules for treated one increased initially, attained the maximum value for particular value of concentration of fillers and decreased. The modulus of treated magnesium hydroxide at

0.14 volume fraction is about 10.01 times higher than that of untreated magnesium hydroxide .The rate of increment in the property with increasing volume fraction of the filler.

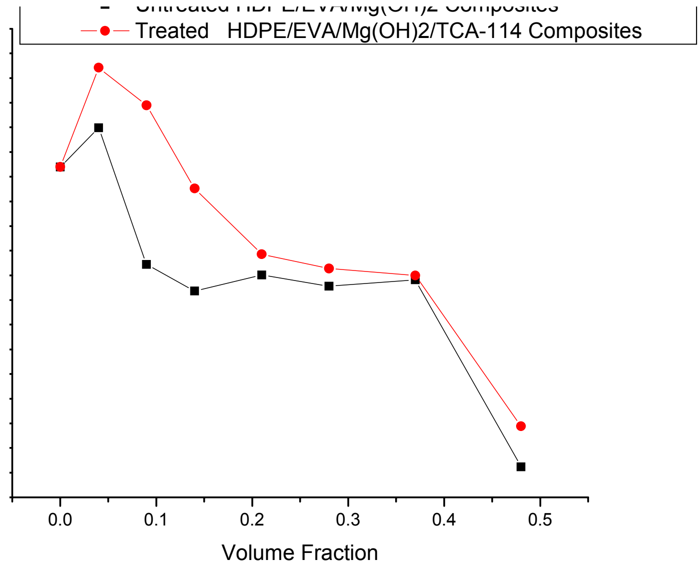

Figure (1): Tensile Strength of the Treated & untreated EVA/HDPE/$Mg(OH)_2$/TCA-114, Composites.

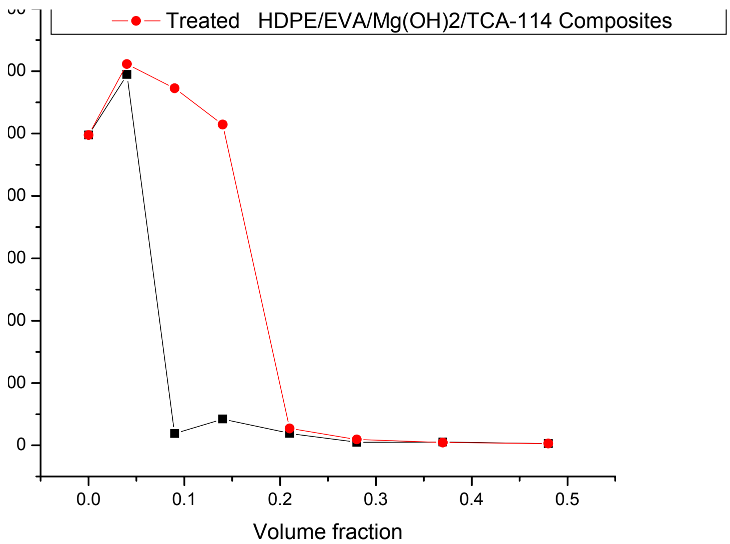

Figure (2): Elongation at break of the Treated & untreated EVA/HDPE/$Mg(OH)_2$/TCA-114, Composites.

Elastics Modulus

Figure 3 shows the dependence of elastics modulus on concentration of treated and untreated filler in HDPE/EVA. It is seen that, Elastics Modulus of both treated and untreated $Mg(OH)_2$ -EVA composite increased on increasing the concentrations of fillers .

The elastic modulus of treated magnesium hydroxide at 0.14 volume fraction is about 2 times higher than that of untreated magnesium hydroxide .The rate of increment in the property with increasing volume fraction of the filler.

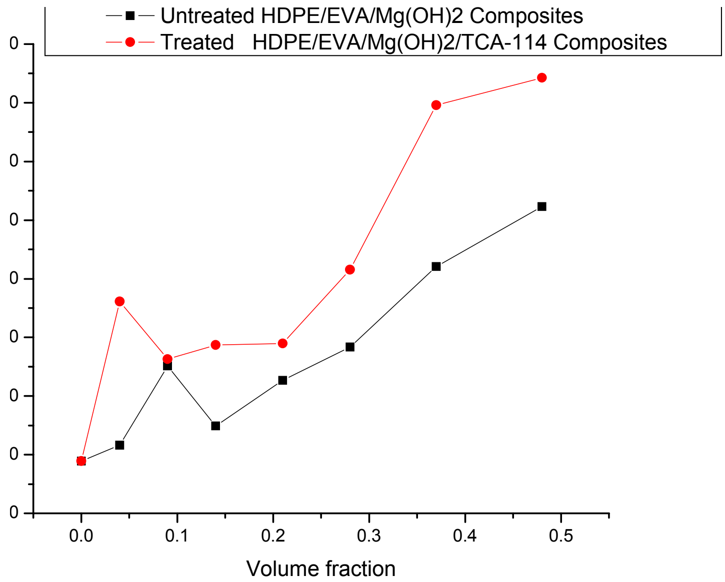

Figure (3): Elastic Modulus of the Treated & untreated EVA/HDPE/$Mg(OH)_2$/TCA-114, Composites.

Hardness

Figure 4 shows the dependence of hardness on concentration of treated and untreated filler in HDPE/EVA. It is seen that, hardness of both treated and untreated $Mg(OH)_2$ – HDPE/EVA composite increased on increasing the concentrations of fillers, with a constant rate of increment for composites containing treated and untreated filler (separately) as evidenced by constant and identical slopes of the lines (figure 4). The hardness of treated magnesium hydroxide at 0.14 volume fraction is about 1.01 times higher than that of untreated magnesium hydroxide .The rate of increment in the property with increasing volume fraction of the filler.

SEM of Composites

The SEM photomicrographs of filler magnesium hydroxide and Titanate coupling agent are shown in plate 1&2. It is clear from these photographs that untreated magnesium hydroxide and Titanate coupling agent show tendency to form agglomerates. SEM of HDPE/EVA/$Mg(OH)_2$ / Composites are shown in plates 3-7 . Untreated composite fracture shows non -adhesive appearance and formation of agglomerates while treated composites show a very uniform distribution, regular, adhesive appearance indicating further enhancement in polymer–filler attachment.

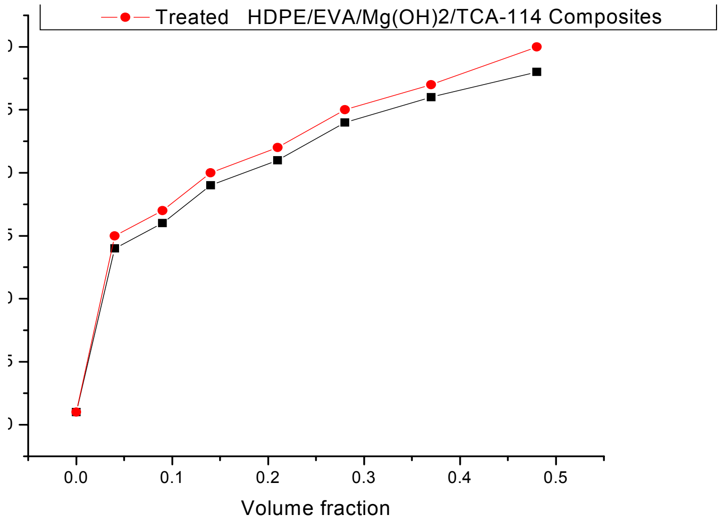

Figure (4): Hardness of the Treated & untreated EVA/HDPE/$Mg(OH)_2$/TCA-114, Composites.

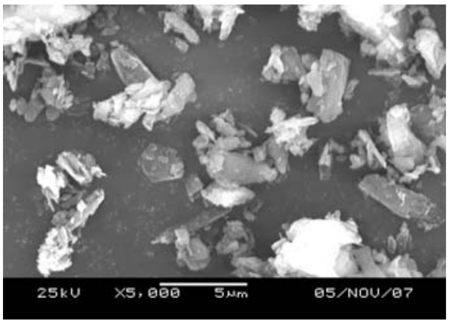

Plate (1): SEM of $Mg(OH)_2$ Powder (2µm).

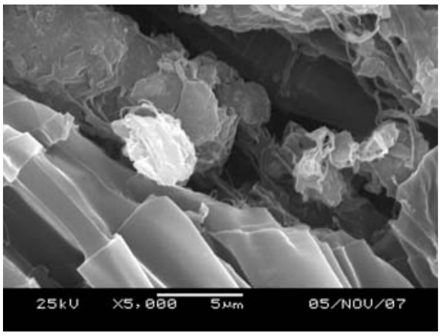

Plate (3): SEM of EVA Composites.

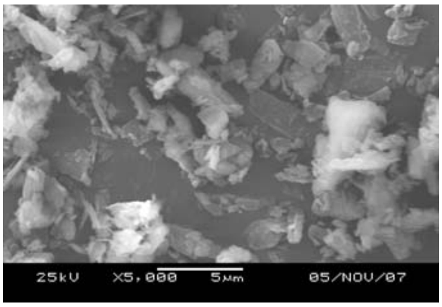

Plate (2): SEM of Mg(OH)2/TCA-114 Composites.

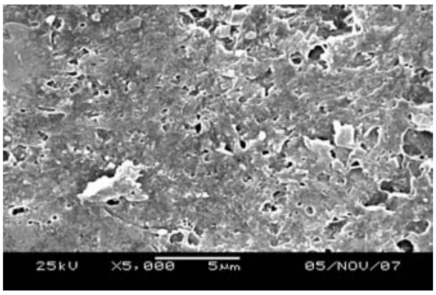

Plate (4): EVA/$Mg(OH)_2$.

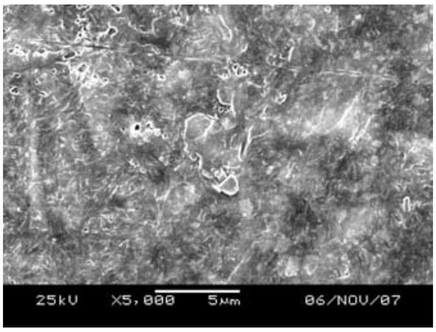

Plate (5): HDPE/$Mg(OH)_2$.

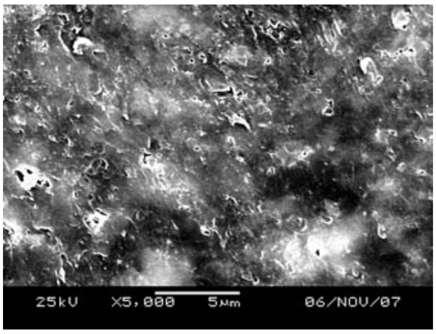

Plate (6): HDPE/EVA/$Mg(OH)_2$ Composites.

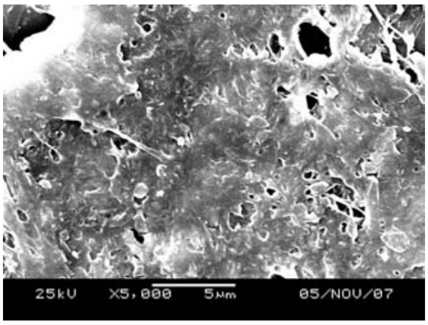

Plate (7): HDPE/EVA/$Mg(OH)_{2/}$ TCA-114 Composites.

CONCLUSIONS

Treated Magnesium hydroxide composites showed improvement in mechanical properties and the mechanism of adhesion due to titanate coupling agent is proposed for Magnesium hydroxide as filler.

The treatment of Magnesium hydroxide with titanate coupling agent has effected magnitudes of (%) elongation at break, tensile strength and elastic modulus and hardness of HDPE/EVA/$Mg(OH)_2$ / Composites. The filler treatment proved to be beneficial by enhancing polymer – filler adhesion as evidenced by SEM study. Considering the cost of the filler and the improvement in properties, the treatment is advisable.

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دراسات في تأثير عوامل الرابط كمواد رابطة في تحسين الخواص الميكانيكية للهيدركسيد المغناسيوم ، أدخلت كحشوه في متعدد البولي إيثيلين عالي الكثافة ومع كوبوليمر اثيلين فينيل – اسيتات

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ملخص :

سجلت في هذه الورقة العلمية دراسات في تأثير عوامل كمواد رابطة في تحسين الخواص الميكانيكية الميكانيكية للهيدركسيد المغناسيوم ، أدخلت كحشوه في متعدد البولي إيثيلين عالي الكثافة ومع كوبوليمر اثيلين فينيل – اسيتات.

المواد الرابطة في شكلها سائل بنسبة 1.5% وأضيفت في معالجة مادة الحشوه هيدروكسيد المغنسيوم .

المادة المعالجة بعد ذلك أعطيت نتيجة عاليه في الخواص الميكانيكية للمتكون متى ما قورنت هذه النتيجة مع المتكون المحتوى فقط على الهيدروكسيد المغناسيوم الغير معالج .

والخواص التي أخذت في الدراسة مثل : استطالة الشد ، معامل القطع ، معامل اللزوجة والقسوه.الخ .

والدعم كان جيد مع ملاحظة من خلال إضافة المادة المعالجة بنسبة 1.5% من المادة الرابطة والملاحظة كانت النتيجة عاليه العلامة بالمقارنة مع الغير معالج كما في الحالة الأولى . ومقارنة الخواص المتكونة من إدخال المعالج والغير معالج من الهيدروكسيد المغناسيوم . وحسمت أن المعالج مع الهيدروكسيد المغناسيوم أعطيت نتيجة أفضل في الخواص الداعمة المتكون (المصنوع) . والخواص المدروسة كانت على النحو التالي : استطالة الشد – معامل القطع – معامل اللزوجة والقسوه ...الخ . واستطالة الشد أعطت علامة في التحسين هي 19.10% ، معامل القطع 9.01%، ومعامل اللزوجة أعطت 100% بينما القسوه أعطت علامة 0.5% وبالتحديد عند جزء من الحجم وهي 0.14% .

كلمات رئيسة : عوامل الربط ، الخواص الميكانيكية للمتكون ، متعدد البولي إيثيلين عالي الكثافة ، هيدركسيد المغناسيوم ، كوبوليمر اثيلين فينيل – اسيتات.